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Construction and use of a Bingham type viscometer

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CONSTRUCTION AND USE
of a
BINGHAM TYPE VISCOMETER

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A thesis, presented to the Department of Chemistry of Union College, in partial fulfillment of the requirements for the degree of Bachelor of Science in Chemistry.

by Harold P. Bodenstab
4/14/43

Approved by Charles G. Hurd.

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Construction and Use of a Bingham Type Viscometer.

Bingham in his book "Fluidity and Plasticity" has described a type of viscometer similar to the Ostwald viscometer but which has the added feature of a variable applied pressure. By changing the pressure the time of flow may be controlled so that a rather viscous liquid will not have an inconveniently long time of flow in a viscometer which is suitable for another liquid. The construction of such an apparatus consists in the assembly of a special viscometer with an arrangement for applying air pressure, measurement of such air pressure, and the placement of the viscometer in a thermostat. Such an arrangement may be seen in the included diagram.

Viscosity measurements are made after filling the viscometer with the correct amount of liquid at the desired temperature. This amount is determined by a mark on the viscometer and the overflow trap. Air pressure is applied to one arm of the viscometer while the other arm is opened to atmospheric pressure. The time of flow for the liquid between two marks is recorded and the air pressure is switched to the opposite arm, opening the first arm to the atmosphere. To facilitate the changing directions of air pressure, two three-way stopcocks are used.

The actual construction of this apparatus was delayed and as yet unfinished for several reasons, among them the unavailability of various parts.

The Apparatus

The thermostat bath must be large enough to contain the viscometer, a thermoregulator, heater, stirrer, and thermometer. Since the viscometer would be about twelve to fourteen inches long, the bath would necessarily be at least twelve inches deep. To reduce radiation losses, a clear Dewar flask should be selected whose diameter is at least six inches.

The stand for the Dewar flask is made from two three-quarter inch plywood squares, eighteen inches on a side. These are separated by four three-quarter inch pipes held by pipe flanges. The length of the pipe is determined by the size of the Dewar flask.

The viscometer is suspended from a bracket, fastened to the top plywood, by a suitable clamp. The clamp and bracket are not shown on the diagram. The relative position of the viscometer in the bath is in the front center to allow it to be seen readily for measurements. Each arm of the viscometer is connected by a loop of rubber tubing and a three-way stopcock to the air pressure reservoir. The two three-way stopcocks are mounted on the front of the top plywood board for accessibility. Filters should be inserted between the stop-cocks and the viscometer to remove any possible dust particles from the air before it enters the viscometer. Glass wool is unsuitable and not recommended as small fibers of glass may break and be blown into the viscometer. A material such as "Lambs' wool" has been used successfully.

The temperature of the bath is controlled by a thermoregulator and a thyratron circuit and measured by a thermometer graduated in at least tenths of a degree. The thermoregulator is shown in detail on the diagram as a mercury-toluene type. Toluene was originally to be used but any other liquid with a relatively high coefficient of cubical expansion may be used, possibly methyl alcohol. The thyratron circuit will control the heater current. The heater might well be a coil type wound on a tube surrounding the stirrer. However a knife type heater is available. This heater should be located near the stirrer to insure a rapid response of the bath to temperature changes. The motor stirrer is a direct drive type connected to the propeller shaft by a rubber connection to eliminate some vibrations. The motor is mounted on a vertical rod from the plywood board. It may be necessary to equip the propeller shaft with two propellers if the Dewar flask bath is too deep.

If radiation losses from the bath are still too high, other precautions may be taken. About half of the bath may be covered with some sort of insulation material leaving only enough space uncovered to see the working part of the viscometer. A cover may also be necessary.

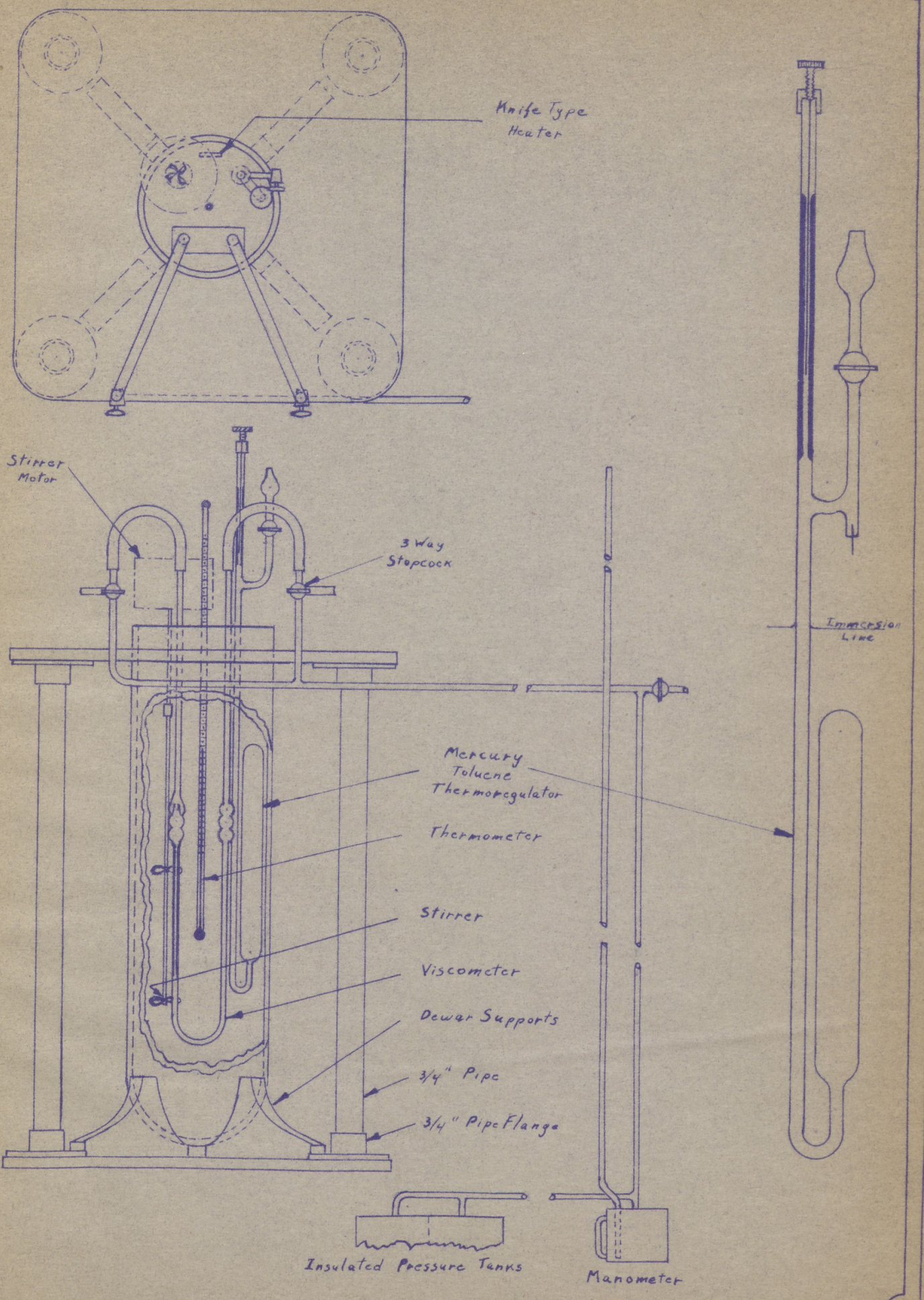
Air pressure is supplied from an insulated reservoir. A large steel tank is preferable if obtainable. Two five gallon

glass carboys may be substituted. The volume is necessary to reduce the error due to a small change in pressure arising from the change in working volume of the viscometer. With ten gallons of air a volume change as great as twenty milliliters is inappreciable. The insulation is obtained from excelsior packed boxes. The reservoir is filled from the air compressor and shut off by a single stopcock. After filling, time should be allowed for the air to come to constant temperature. In going from the relatively high pressure of the compressor to the low pressure reservoir, a Joule-Thomson effect is involved.

The manometer is constructed as indicated on the diagram. The manometer tube extends close to the bottom of the manometer reservoir. A gauge tube forms a closed circuit from the bottom of the reservoir to the top. It shows the level of the manometer liquid from which the height of the pressure column is measured. The gauge tube and the manometer pressure tube should be of the same diameter to eliminate the capillary action effect. The materials for construction would be best if all glass, but this involves some difficult glass blowing. As a substitute, a cylindrical metal reservoir connected by rubber tubing to the glass tubes may be used. The manometer tube should be large enough in diameter to make the meniscus partially flat. This will make it easier reading. The size of the manometer reservoir

is determined so that a small variation of the height of the pressure column will make no effective change in the position of the liquid level in the gauge tube. It may be easily shown that the ratio of the change in heights of the two columns is inversely proportional to the ratio of the squares of the radii of the pressure tube and the reservoir, $\frac{\Delta H}{\Delta h} = \left(\frac{r}{R}\right)^2$ where ΔH and R are the change in height and radius of the reservoir and Δh and r are the corresponding dimensions of the pressure tube. Thus, if the change is to amount to as much as 1%, the larger radius is ten times the smaller radius. To be specific, if the larger tube is one half inch in diameter, the reservoir should be at least five inches in diameter. The height of the reservoir should be great enough so that its volume is at least equal to the volume of the pressure tube. If the pressure tube is six feet high, the cylindrical reservoir should be at least 0.72 inches or practically one inch.

As the pressures dealt with will not be excessive, water will make a good liquid for the manometer. A small amount of a substance to reduce the surface tension, thereby flattening the meniscus, without changing the density appreciably is offered as a suggestion to be tested.


Knife Type Heater
Stirrer Motor
3 Way Stopcock
Immersion Line
Mercury Toluene Thermoregulator
Thermometer
Stirrer
Viscometer
Dewar Supports
3/4" Pipe
3/4" Pipe Flange
Insulated Pressure Tanks
Manometer

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Tricyclohexyl Aluminum

by Harold P. Bodenstab

Approved by F. C. Schmidt

Tricyclohexyl Aluminum

In a brief search of chemical literature no mention of tricyclohexyl aluminum was found, but compounds which contain similar constituents have been prepared. Grazuly[1] describes the preparation and properties of triphenyl aluminum. This compound is prepared from diphenyl mercury and aluminum sheets in an inert atmosphere of either hydrogen or nitrogen. Gruttner[2] reacted cyclohexyl magnesium bromide with bismuth tribromide in an ether solution and an atmosphere of carbon dioxide to obtain probably tricyclohexyl bismuth. This was quickly oxidized by contact with air. Gilman[3] gives as a general preparation of triaryl organo aluminum the reaction of aluminum and the diaryl mercurial compound.

The method of Grazuly and Gilman was at first thought well applicable to the preparation of tricyclohexyl aluminum. This method of reacting aluminum with the dicyclohexyl mercury was abandoned when it was found that according to Gruttner[4] dicyclohexyl mercury melts at 78-9° C giving a clear liquid which deposits mercury in a few seconds. This may be too unstable for the reaction with aluminum unless a way can be found to make the reaction proceed below 78° C.

Experimental

The preparation of tricyclohexyl aluminum by means of the Grignard compound and aluminum trichloride was chosen as a first attempt. Cyclohexyl bromide was made from cyclohexyl alcohol and hydrogen bromide and dried with anhydrous calcium chloride for over a week. The bromide was distilled and the fraction distilling between 160-165° C collected. The Grignard was made according to the preparation of Gilman[5] using 75 cc. (0.6 mole) of cyclohexyl bromide, 22. gm. (over 0.9 mole) magnesium and about 200 cc. of dry ether. The reaction was run in an atmosphere of nitrogen and stirred by shaking occasionally.

Assuming the 80% yield which Gilman states as being atainable would give about 0.48 mole of cyclohexyl magnesium bromide. It requires three moles of the Grignard to one mole of aluminum to form the tri-substituted aluminum compound. To cause the tri-substituted compound to form, an excess of the Grignard is needed. On this reasoning less than one-third of 0.48 or 0.16 makes of aluminum tri-chloride was needed, or less than 22. gm. of anhydrous aluminum tri-chloride was used, which was disolved in 120 cc. of dry ether forming a black colored solution.

The aluminum tri-chloride ether solution was added to the Grignard reaction compound over a fifteen minute period. The reaction was vigorous, forming a solid product.

Identification of this reaction product has not been completed. A rough aluminum analysis or some of the reaction product which had been dried under vacuum gave an average

of 11.2% aluminum. The theoretical percentages of aluminum are 9.7% in tricyclohexyl aluminum and 11.7% in dicyclohexyl aluminum chloride. This may show the reaction product to be a mixture of these two compounds but it is not necessarily valid because in all likelyhood there is some unreacted aluminum tri-chloride remaining in the unpurified compound.

Several solubilities were tried, the reaction product being partially soluble in benzene and ether and practically insoluble in cold carbon tetrachloride. A solubility test in acetone seemed to indicate a reaction as a gas was evolved and the solution became yellow. If excess of the product is added to the acetone solution the solution becomes colorless.

Purification of the reaction product was attempted by extraction with dry benzene in a Soxhlet extraction apparatus. Care must be taken so that the amount of benzene in the reservoir does not become too low or that the concentration of the extracted product become too high in the benzene reservoit as decompositions of the product will result. The extraction has not been completed or the extraction product identified.

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